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INFLUENCE OF SEGREGATION ON THE RHEOLOGICAL BEHAVIOR OF CEMENT GROUTS DETERMINED BY COAXIAL CYLINDER RHEOMETER

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ABSTRACT

This work aims to study the phenomenon of segregation during the rheological characterization tests and influence of this phenomenon on the rheological behavior of cement grout. Thanks to a new methodology composing a specific protocol and several density measurements in the sheared volume of grout, this work helps to identify the segregability of cement grouts under shearing during rheological tests. We have studied many mix-designs of grout by varying the W/C ratio and the dosage of superplasticizer. The relationship between the specific rheological behavior of segregated grout and the evolution of solid fraction in the sheared volume in the rheometer has been researched.

The overall results allow to determine the influence of different mix designs and to understand thoroughly the impact of segregation on the rheological behavior of the cement grout during the characterization rheology due to shearing.

KEYWORDS: Rheology, Cement Grout, Segregation, Rheometer, Density, Super Plasticizer & W/C Ratio

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Original Article

1. INTRODUCTION

The rheology is one of the most important properties of cement grout and cementitious material. It affects not only their quality in fresh state, but also in hardened state(Banfill, 2003). In fact, the characteristics of hardened concrete, for example, often depend on the characteristics of fresh concrete during transport, installation and finishing.

Generally, the rheological behavior of cementitious grouts can be determined accurately and completely by using rheometers. Amongst several types of apparatus, rotary rheometers with coaxial cylinders are the most frequently used apparatus for the rheological characterization of cement grouts, due to itshighaccuracy and capabilityto determine the flow curve, regardless of the rheological behavior of the material (Kamal H. Khayat, 2002; Phan et al., 2006; Roussel & Le Roy, 2005). The rheological characterization is only invalid, when the material retains its homogeneity and does not present any segregation or bleeding(Schierbauni, 1964). In fact, during the rheometric measurement with a coaxial cylinder rheometer, under the action of shear, the dynamic stability of the grout may be compromised, which deteriorate the quality and reliability of measurements. Indeed, even the stable grout at rest can become segregated under shearing(Boucenna, et al., 2002).

However, very few studies are focused on the dynamics segregation phenomenonof the grout in the rheometer and no reliable methodology for assessing has been proposed so far. In this study, we gained insight into

the segregation phenomenon of cement grout under shearing. Also, by using a new methodology of characterization, we clarify the influence of the segregation of a grout on its rheological behavior.

2. MATERIALS AND EXPERIMENTAL METHODS

2.1 Materials

All grouts of the study are prepared with a Portland cement CEM I 52.5N CP2, according to the European Standard EN 197.1 (N. E. 197-1,) and the French standard NF P 15318(‘N. P15-318.). Table 1 presents the main chemical and physical properties of the cement.

Table 1: Chemical and Physical Properties of Cement

Composition	Value	Unit
C_3S	70	%
C_2S	8	%
C_3A	9	%
SO_3	2.9	%
Na_2O equivalent	0.54	%
Physical and Mechanical Properties		
2 days compressive strength	30	MPa
28 days compressive strength	60	MPa
Blaine specific surface area	3790	cm²/g
Density	3.15	g/cm^3
Water demand for standard paste	28	%
Final setting time	2.30	hh.mm

A new generation modified polycarboxylate is used as super plasticizer; allowing efficient dispersion of cement particles and improving grout’s flowability for extend duration. Table 2 present the main properties of this superplasticizer.

Table 2: Main Properties of Super Plasticizer

Properties	Value	Unit
Density	1.07 ± 0.02	g/cm^3
pH	6.5 ± 1.5	
Cl^-	< 0.1	%
Na_2O equivalent	< 2	%
Dry material content	30 ± 1.5	%
Recommended dosage (/cement)	0.2 - 2.0	%

2.2 Rheological Protocol and Density Measurements

The mixing of grouts was carried out on 800 ml mixture with a blender revolving at 700 rpm during 7 min. We verified that this duration was enough to guaranty full homogenization of the mixture without any segregation at the end of the mixing for all studied grouts.

We have developed a new methodology to characterize the rheological behavior and the potential segregation of cement grouts(Hoanget al., 2015). This methodology uses a rotational rheometer with coaxial-cylinder geometry (Couette type) and a specific protocol composing of two shearing cycles with various steps of shear rate between 0 and 500 s^{-1}(Figure 1) and two values of bottom gap (1mm and 10mm) (Figure 2). Density measurement of the upper and the lower part of the grout’s sheared volume in the rheometer were carried out at different characteristic times of the protocol (listed from 0 to 4 in Figure 1), and compared with rheological behavior of grout during shearing.

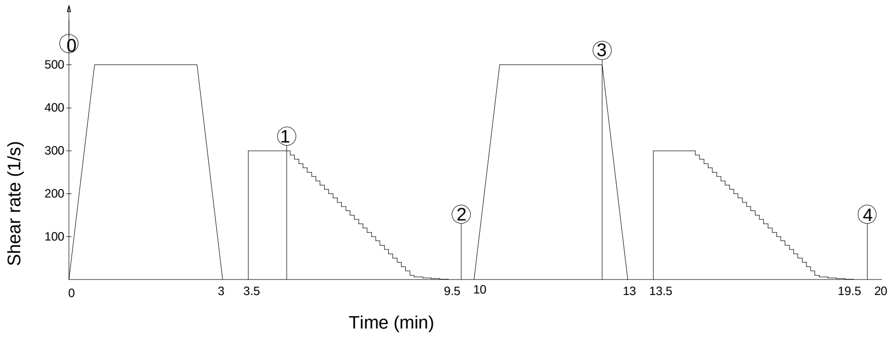


Figure 1: Specific Protocol for Rheological Characterization of Grouts

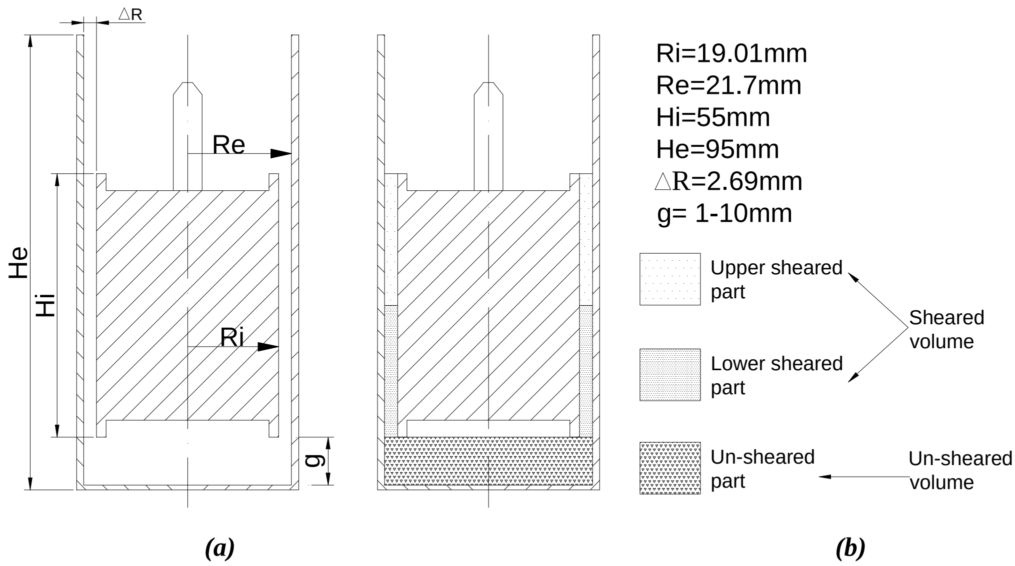


Figure 2: (a) Geometrical Characteristic of used Coaxial Cylinder

(b) Designation of different Volumes of Grout in the Coaxial Cylinder

The methodology was successfully applied and validated on a *stable grout* without any super plasticizer, and an *unstable grout* with high amount of superplasticizer. It has been shown that the unstable grout presents a special behavior, which depends strongly on the height of bottom gap. In detail, a characteristic **peaks of shear stress** are observed at the beginning of high shear rate step (500 s^{-1}) in 2^{nd} cycle, and the **flow curve of segregated grout appearsmore viscous when the bottom gap is low (1 mm)**. This special behavior is not observed in the case of stable grout (Hoang et al., 2015).

3. EXPERIMENTAL RESULTS AND ANALYSIS

Considering the cement grout as solid grains of a suspension, in terms of rheology, it describes commonly as the solid volume fraction rather than density. We, therefore, switch the density measurements of the grout over the fraction by the following equation.

$$\phi = \frac{\rho_G - \rho_W}{\rho_C - \rho_W} \qquad (1)$$

Where, ϕ is the solid volume fraction of grout, ρ_G, ρ_W, ρ_C are the density of grout, cement and water, respectively.

To estimate the segregation of the grout at the end of first cycle, we then determined the decrease of the solid fraction in **the upper part of the sheared volume** by the equation 2. This part is the most representative of the segregation phenomenon, and will be subsequently used to assess the potential for segregation of the grout (Hoang et al., 2015).

$$\Delta\phi_{0-2} = \frac{\phi_0 - \phi_2}{\phi_0} \times 100\% \quad (2)$$

Where ϕ_0 is the initial solid volume fraction of the slurry (after mixing), and ϕ_2 is the solid volume fraction at the end of the first cycle.

To study the influence of the W/C ration and superplasticizer on the potential segregation and rheological behavior of the grout, we carried out the experimental measurements on four grout sets corresponding to four different W/C ratio (0.35; 0.40; 0, 45 and 0.50) with the dosages of superplasticizer between 0% and 1%.

3.1 Influence of the W/C Ratio

It is obvious that the W/C ratio (or the solid volume concentration) significantly affects the stability of the grout. To highlight the influence of the W/C ratio, it is useful to begin from the simplest cementitious mixtures: cement grout compounds cement and water without any superplasticizer.

As show in Figure 3, the decrease of the solid fraction, $\Delta\phi_{0-2}$ obtained with the gap 10 mm is systematically higher than that obtained with the gap 1 mm. The difference between the two gaps progressively increases with W/C ratio. We observe, moreover, that the $\Delta\phi_{0-2}$ increases with W/C ratio in both value of the gap. This increase becomes great from the W/C ratio 0.45. We think that this value of W/C ratio corresponds to a change of segregation regime of the grout.

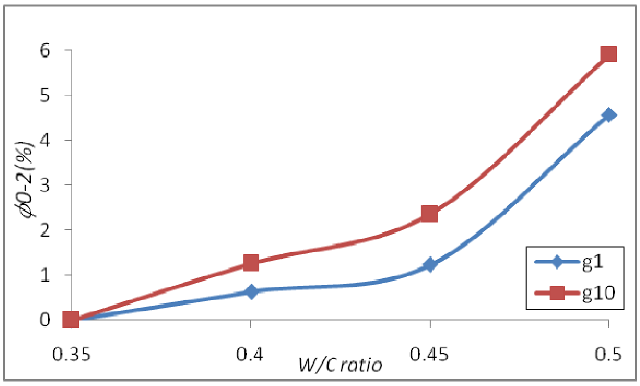


Figure 3: The Decrease of the Solid Fraction of Grout without any Super Plasticizer

To confirm this change, we then compared the behavior of the grout with W/C 0.45 and the grout with W/C 0.5 (Figure 4). The comparison of the rheological behavior of the two grouts reveals that the grout with W/C 0.45 presents a quasi-independent behavior of the gapduring the rheological measurement. In this case, the flows curves obtained are very close in both bottom gaps only in the 1st cycle. Whereas, the shear stress with the gap 1mm is higher than that with the gap 10 mm in the case of the grout withW/C ratio 0.50. In addition, this shear stress difference is higher in the second shearing cycle than in the first.

The experimental result also shows that with the very high W/C ratio, the grout without any superplasticizerlosesits homogeneity. This phenomenon is also demonstrated in the studies of Houls by (Houls by, 1990) and Perrot et al (Perrot et al., 2012). However, in this case, no stress peaks were observed in the second pre-shearing step at

500 s^{-1} but a big difference of the shear stress between the measurement of gap 1 mm and gap 10 mm. This phenomenon also highlights the fact that the disintegration of segregated grout into the rheometer is strongly attenuated for grout, without any super plasticizer (Hoang et al., 2015).

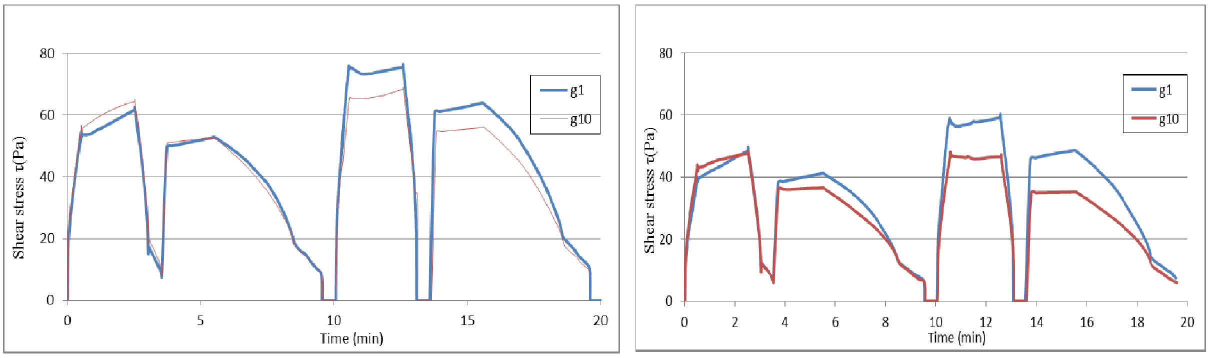


Figure 4: Variation of the Shear Stress of the Grout with (a) W/C 0.45 and (b) W/C 0.5 during the Rheological Characterization Using 1 mm and 10 mm Bottom Gap

To understand thoroughly the influence of the solid fraction on the segregation of cement grout, we continue with a series of tests over the previous four groutswith 0.4% of superplasticizer. In Figure 5, the decrease of the solid fraction of grout is plotted as the function of the W/C ratio.

We observed a point of inflection on the variation curve of $\Delta\phi_{0-2}$ between W/C 0.4 and 0.45. We thought this inflection point corresponds toa value of solid fraction, which differentiates the physical behavior of the segregation of the grout. To better understand this behavior change, we compare the difference between the shear stress of the grout W/C 0.4 and 0.45 (Figure 6).

We noted that because of segregation, the value of shear stress of both grouts depends on the gap. However, thedifference of stress between two gapsand the peak intensity of second pre-shearing step at 500 $s^{-1}$are more obvious in the case of the grout of W/C ratio 0.45.

In addition, at the first pre-shearing step, we saw a steady increase in the shear stress in the case of grout W/C 0.4. This variation of the shear stress is the result of the rebuilding phenomenon of the grout, because of its high volume fraction of cement(Ó. H. Wallevik and J. E. Wallevik, 2004).

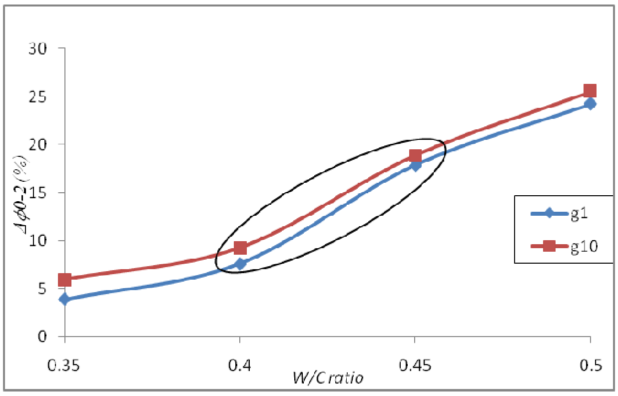
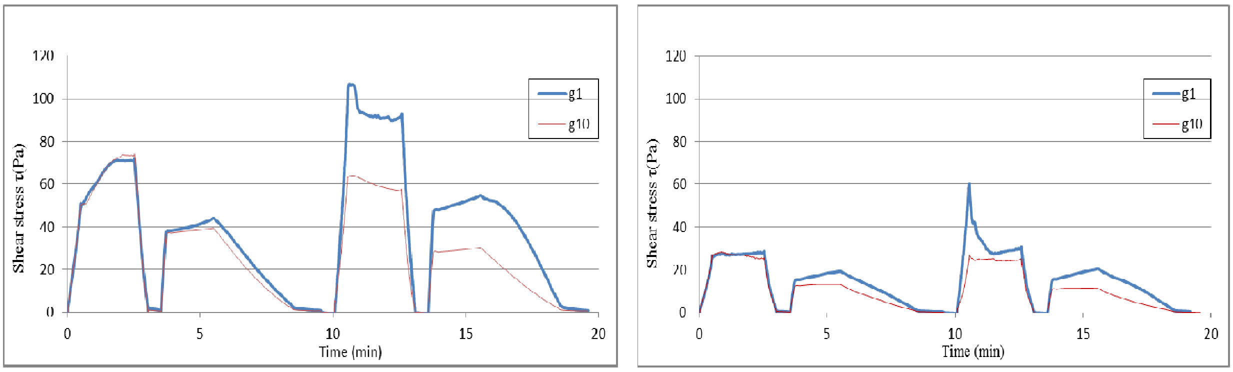


Figure 5: The Decrease of the Solid Fraction of Grout with 0.4% of Super Plasticizer



(a) **(b)**

Figure 6: Variation of the Shear Stress of the Grout with 0.4% Super Plasticizer with (a) W/C 0.4 and (b) W/C 0.45 during the Rheological Characterization Using 1 mm and 10 mm Bottom Gap

To sum up, we can say thatthe segregation of cement grout is more important when the volume concentration of solid particles is low, which reduces the attractive interactions between particles and promotes the defloculation of the grout'sstructure. This result correspond well with other studies of Radocea(Radocea, 1992), Peng and Jacobsen (Peng & Jacobsen, 2013; Peng, et al., 2013) and Perrot *et al* (Perrot et al., 2012).

3.2 Influence of Superplasticizer

The dosages of superplasticizer strongly influence on the stability of cementitious grout. When the concentration of superplasticizer is high, the attractive interactions are decreasedand the particles are deflocculated.

The experimental result confirms that thesegregation canoccur even at very low dosage of superplasticizer. It is also worth to be noted that, ata high enough dosage of superplasticizer, the segregation of the groutno longer changes indicating a form of saturation. This phenomenon is also shown in the study of Neubauerand Yang(Neubauer, Yang, & Jennings, 1998; Yang, Neubauer, & Jennings, 1997).

To analyze further the results of the previous tests on the solid fraction, we then studied variations in the shear stress.

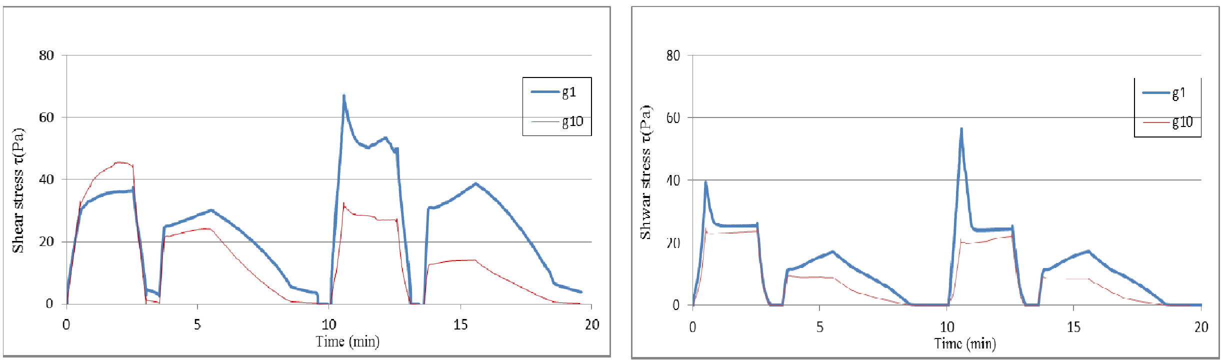


Figure 7: Variation of the Shear Stress of the Grout with W/C 0.45 Containing (a) 0.2% Superplasticizer and (b) 1% Superplasticizer during the Rheological Characterization Using 1 mm and 10 mm Bottom Gap

The first observation on the rheological behavior of the grout is that, the shear stress as function of shear rate decreases with increasing dosage of superplasticizer. This is inherent with the dispersant role of the superplasticizer.

The second observation on rheological behavior of the grout is, with the increase of the dosage of superplasticizer, the shear stress measured for the different shear rates depends on the bottom gap of the rheometer. Indeed, the higher the grout is segregated, the bigger difference between theshearstress measured with the gap of 1 mm with the gap of 10 mm is, and the higher intensity of the peak stress to the second pre-shearing step is as shown in Figure 7.

4. CONCLUSIONS

In conclusion, by analyzing variations in solid fraction and the shear stress of different groutsand different values of bottom gap, this work revealed that theW/C ratio and dosage of superplasticizer have an obvious impact on the potential segregation of cement grout under shearing, and this phenomenon also decide the rheological behavior …

It is noted that the increase of the dosage of superplasticizer and the decrease of the initial solid fraction solid boost the potential segregation. The results show that the stable grout presents a quasi-independent behavior of the gapduring the rheological measurement by thecoaxial cylinder rheometer. On the contrary, the shear stress with the gap 1mm is higher than that with the gap 10 mm in the case of thesegregated grout. Furthermore, we also identify some special rheological behaviors, for example, the peaks of shear stress, the rebuilding phenomenon. Thus, it is easily to distinguish the intensity of segregation of a cement grout (more or less segregated) and verify thereliability of rheological measurement.

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